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In situ Characterization of Trans-laminar Fracture Toughness using X-ray Computed Tomography

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Abstract

The change in the critical strain energy release rate as damage evolves, known as the R-curve, is of crucial importance to the understanding of fracture behaviour. The examination of damage evolution ahead of the crack tip in order to determine accurately the crack increment is key for the determination of the R-curve. Conventional *in situ* methods such as optical measurements only examine the specimen surfaces. X-ray Computed Tomography (CT) offers satisfactory image quality, but conventional CT scanning requires the removal of the specimens from the test machine. If no dye penetrant is used, the specimens can be re-loaded, but some important information will be missing such as the early load drops corresponding to damage initiation. If dye penetrant is used, the specimens can no longer be re-tested. In contrast, *in situ* CT scanning can capture the detailed damage states ply-by-ply while the specimen is loaded and diminish the need for multiple specimens. *In situ* characterization of trans-laminar fracture toughness of composites using CT has not been attempted in the past. It has been proven successful in this research, and shown that a partial R-curve can be constructed with a single Extended Single-Edge-notch Tension (ESET) specimen.

Keywords: Fracture, Non-Crimp Fabric (NCF), R-curve, Computed Tomography (CT)

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1. Introduction

The upsurge in use of advanced composites in airplane primary structures has promoted much research lately, however, the need for further knowledge about their failure mechanisms, especially translaminar fracture is still apparent. The change in the critical strain energy release rate as damage evolves forms a fracture resistance curve, known as the R-curve, which is of crucial importance to the understanding of fracture behaviour. To determine the R-curve, the measurement of crack increment, Δa, is critical and has proven challenging. Various non-destructive methods have been tested including visual inspection, Digital Image Correlation (DIC) and X-ray Computed Tomography (CT). Pinho et al. [1] carried out fracture toughness characterization of carbon/epoxy laminated composites using compact tension and compact compression specimens. An *in situ* optical crack measurement approach was employed by means of a photogrammetry system (Aramis), which monitors the crack propagation by recording the strain field near the crack tip, but only on the specimen surface. Zobeiry et al. [2] have shown the capability of DIC in damage characterization using carbon/epoxy laminates using an Over-height Compact Tension (OCT) configuration, again based on the strain measurements on the specimen surface. However, damage in composite laminates is different in different plies and hence surface measurements are not reliable. In contrast, it was found that X-ray CT yielded accurate results when assessing the internal damage states of the specimens [3]. X-ray CT scanning of polymer composites has been studied extensively as recently reviewed by Garcea et al. [4]. However, there are issues that are intrinsic to X-ray CT which cannot be neglected and need to be addressed in order to better utilize the penetrating characteristic of CT scans. Conventional X-ray CT scanning requires the specimen to be removed from the test machine, and sometimes the application of a dye penetrant can aid in visualizing nearly closed cracks, both of which prohibit a streamlined and efficient data collection process, not to mention the higher uncertainty introduced by using multiple specimens. *In situ* CT scanning is an attractive tool for experimental mechanics as reviewed by Buffiere et al. [5]. It is a viable alternative and substitution for conventional X-ray CT as it overcomes the problems posed by the latter while still maintaining the advantages of CT scans. *In situ* CT scanning, as its name suggests, has the CT scanning process take place during the testing without the need to remove the specimen from the test machine. Because of the nature of the *in situ* tests, the specimen is under load during CT scanning, which keeps cracks open, making it easier to see the damage. Also, with *in situ* tests multiple specimens are not necessary for examining the damage states at different load levels. However, the *in situ* loading devices within X-ray CT scanner can limit the maximum resolution for imaging [4]. On the one hand, the size of the specimen needs to be small as the specimen needs to be as close as possible to the X-ray source. One the other hand, the specimen cannot be placed too close to the X-ray source e.g. due to the presence of a tube surrounding the specimen [5], and the resulting lower resolution compromises crack detectability [4]. Wang et al. [6] have demonstrated the capabilities of *in situ* Synchrotron Radiation Computer Tomography (*in situ* SRCT), in which *in situ* SRCT was used to capture the internal damage of short carbon fibre/epoxy composites. The focus was to improve the image quality but not to characterize material properties. Hong et al. [7] compared the results provided by *ex situ* CT and *in situ* synchrotron X-ray radiography scans for fracture mode characterization in a single-edge-notched specimen under tensile loading. They reported that delamination failures interacting with longitudinal and transverse cracks were not detected by *in situ* X-ray scans due to the limited field-of-view but were detected by *ex situ* CT scan. Hence the conclusion was drawn that *ex situ* and *in situ* scans are complementary and both are required for a comprehensive understanding. The current study challenges that conclusion and aims to demonstrate the capability of *in situ* CT scans and the corresponding calculations that can be made as a result.

An *in situ* CT scan method is used to assess the detailed damage states and crack growth in two Extended Single Edge Notched Tensile (ESET) specimens as per the ASTM E1922 standard [8]. Accurate measurements of crack increments in the 0° plies at every ply during various stages of the crack propagation process were obtained *in situ* with a single specimen, which has not been done in the past. A partial resistance curve (R-curve) was directly constructed *in situ* for the first time using the CT scan measured crack increments. The fracture energy value reported in the previous literature approximately falls on the current R-curve. This research provides a quantitative measurement of the R-curve in these quasi-isotropic laminates through accurately measuring the crack increments *in situ*, and demonstrates the advantages of the *in situ* CT scanning technique for the characterization of fracture properties such as trans-laminar fracture toughness and crack increments.

2. Experimental Set-up

The specimens used for the current study are in strict accordance with the ASTM E1922 standard which details the method for calculating the fracture toughness of laminated specimens. Figure 1 a) shows the dimensions of the specimen. A test panel was manufactured at Japan Aerospace Exploration Agency (JAXA) through Vacuum assisted Resin Transfer Moulding (VaRTM). The material used was bi-axial carbon Non-Crimp Fabric (NCF) made from STS-24k fibres by SAERTEX Co. KG, and the epoxy resin

XNR6809/XNH6809 was manufactured by Nagase Chemtex Co. The stacking sequence used was $[(45/-45)/(0/90)]_{2s}$. The nominal laminate thickness was 2 mm, compared with the measured average value of 2.18 mm. Specimen geometries, including the notch, were cut from a large panel using 1 mm carbide end mills on a Computer Numerical Controlled (CNC) router.

The ESET specimens were loaded on to the test jig inside a polymer tube, loading pins were inserted into the two holes at each end of the specimen, as shown in Figure 1 b). The whole loading tube assembly was then installed on the test jig and was brought closer to the radiation source during CT scanning, as shown in Figure 2 c). The CT scanner used was a Shimazu's inspeXio SMX-225CT FPD HR with a customized *in situ* test rig. The *in situ* CT scans were conducted at JAXA. The test was displacement controlled.

During the initial linear loading phase, *in situ* CT scans were performed at regular intervals while the tests were paused, and the displacement was kept still. During each pause an *in situ* CT scan was carried out. After the initial linear loading phase, a scan was carried out as soon as a load drop occurred.

3. Test Results and Analysis

Two specimens were tested, Figure 2 shows the load and displacement graphs for these two tests. The following points can be deduced based on the graph:

- An approximately linear load-displacement relationship is apparent prior to the first load drop.
- A number of small incremental load drops were present before the catastrophic failure.

- Peak load and catastrophic failure did not occur at the same time, catastrophic failure occurred after the peak load, which corresponds to the compressive failure of the rear end of the specimen.

Each pause location indicates that an *in situ* CT scan was carried out. For every scan, a set of images through the material thickness were obtained. Figure 3 shows a set of typical *in situ* CT scan images for each ply orientation. It should be noted that this set of images was taken from Specimen #2 at 1.12 mm of displacement, which corresponds to the crack state just after the peak load. Based on these images, the damage state in the specimens are clearly visible. For example, in Figure 3 a), the crack in the 0° ply is clearly visible with nearly no splitting. Whereas in all of the 90°, -45° and 45° plies, limited fibre fracture could be observed, and damage mainly consisted of splitting, especially for the 90° ply where damage was splitting only. No obvious delamination was observed except near the specimen surfaces in the current VaRTM processed NCF laminate.

Fracture toughness values are calculated based on the equation in ASTM E1922 [8]:

$$K_C = \frac{P}{B\sqrt{W}}\alpha^{\frac{1}{2}}(1.4+\alpha)[3.97-10.88\alpha+26.25\alpha^2-38.9\alpha^3+30.15\alpha^4-9.27\alpha^5\,]/(1-\alpha)^{\frac{3}{2}} \quad (1)$$

where, K_C = fracture toughness in MPam$^{1/2}$; P = Failure load in MN; $\alpha = {}^{a}/_{W}$; a = effective crack length in m; B = specimen thickness in m; W = specimen width in m.

To convert fracture toughness to fracture energy, the following equation was employed assuming plane stress:

$$G_C = \frac{{K_C}^2}{E} \qquad (2)$$

where G_C = fracture energy, in kJ/m^2; K_C = fracture toughness, in MPa·m$^{1/2}$; E = Young's modulus of the quasi-isotropic laminate, in GPa.

In order to obtain an R-curve, knowledge of the crack length is crucial. With the aid of *in situ* CT scan images, crack increments can be determined accurately. As discussed in [9], the crack length can be defined in different ways. Here, an effective crack length, a, based on the average length of fibre breakage in the 0° plies (referred to as crack length in the remainder of this study) is used for the fracture toughness calculations. This crack length does not distinguish between a through-the-thickness crack and a damage process zone, but it is easy to track throughout the experiment. Each ply's crack increment is measured from the *in situ* CT scan images by using VOLUME GRAPHICS's visualization software VG Studios Max, and added to the initial machined notch length, a_0. There are in total four layers of 0° plies in each specimen, and the effective crack length is calculated as the average crack length for each pause location in Figure 2.

Each *in situ* CT scan was carried out right after the occurrence of a load drop to determine the crack increment Δa, and hence the effective crack length, a (Δa plus the initial machined notch length, a_0). It is assumed that the effective crack length does not change at the load drop, which is in accordance with [9] which implied that the load drops corresponded to the sudden fracture of the ±45° plies rather than extension of the crack in the 0° plies. The failure load P used for each fracture toughness calculation is the value at the load drop.

The fracture energy of the specimen at each pause location is determined and thus the relationship between fracture energy and crack increment can be established. In Figure 2 it can be seen that the pause locations are spread across the whole displacement range, however, meaningful information can only be extracted from a very limited displacement range after the initial crack extends and before the ultimate failure. As no effective crack propagation takes place during the linear loading phase nor after the major catastrophic load drop caused by compressive failure at the specimen rear end (at 1.3 mm POD in Specimen #1 and 1.2 mm POD in Specimen #2 in Figure 2), only the pause locations which lie between approximately 1 to 1.3 mm POD are valid for constructing the partial R-curve. Based on these results and assumptions, the partial R-curve could be established, as shown in Figure 4. It should be noted that the last points on the R-curves were taken when compressive failure also occurred, so their values might be underestimated. The following findings can be extracted from the graph:

- Fracture energy, G_C, increases with the crack increments.
- A plateau is not reached in the partial R-curve, but there were insufficient measurements prior to compressive failure at the rear end of the specimen.
- Maximum value of fracture energy is 139 kJ/m^2.

4. Discussion

The *in situ* CT scan images are compared to the previous CT scan images from interrupted tests in Figure 5 [10]. Different from the conclusions in Hong et al. [7] which compared *in situ* synchrotron X-ray against *ex situ* CT scan with dye penetrant, the quality of the current *in situ* CT scans was found to be as good as the conventional CT scans of the previous interrupted specimens with dye penetrant.

It has been demonstrated that an R-curve exists and can be measured by using a robust test method. In this paper, the ASTM E1922 standard [8] has been used and both its validity and limitations considered. The ASTM E1922 standard is capable of generating a partial R-curve from *in situ* CT scans, which agrees well with the approximate value of 85 kJ/m^2 reported in [10] at about 3 mm effective crack increment in the 0° plies based on the previous interrupted OCT tests and conventional CT scans. Furthermore, the observed R-curve behaviour is also consistent with the previously observed stable fracture propagation after fracture initiation in the full-size stiffened panel reported in [10], which was postulated to be due to the existence of an R-curve [10].

5. Conclusions

The current research makes use of *in situ* CT scanning for the characterization of translaminar fracture toughness for quasi-isotropic Non-Crimp Fabric (NCF) carbon/epoxy laminates for the first time. With the aid of *in situ* CT scanning, accurate crack increment measurements which have proved to be extremely challenging in the literature were made from a single specimen. Using these measured crack increments based on the fibre breakage length in every 0° ply, a partial R-curve was constructed in accordance with the method established in ASTM E1922 [8], but a full R-curve cannot be draw due to compressive failure at the specimen rear end. The experimentally obtained R-curve was compared against the previously reported fracture energy value for initial fracture propagation reported in [10]. A good agreement with the *in situ* CT scan results was achieved in terms of the fracture toughness value at 3 mm effective crack increment, supporting the validity of the current method.

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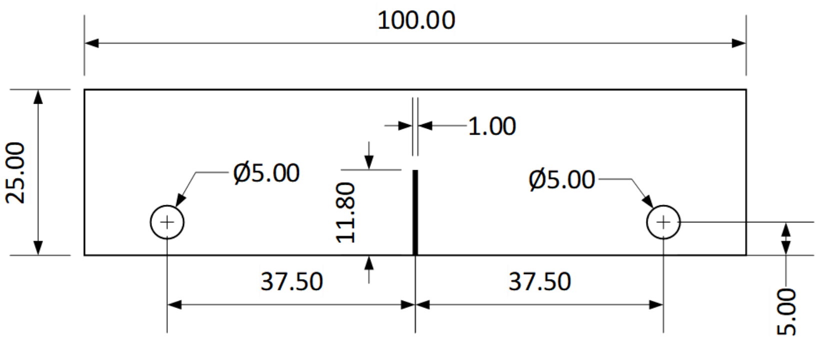

a) *In situ* CT scan specimen geometry and dimensions, based on ASTM E1922 [8]

b) Test rig assembly

c) Test jig set-up for scanning

Figure 1. *In situ* CT scan specimen and test set-up.

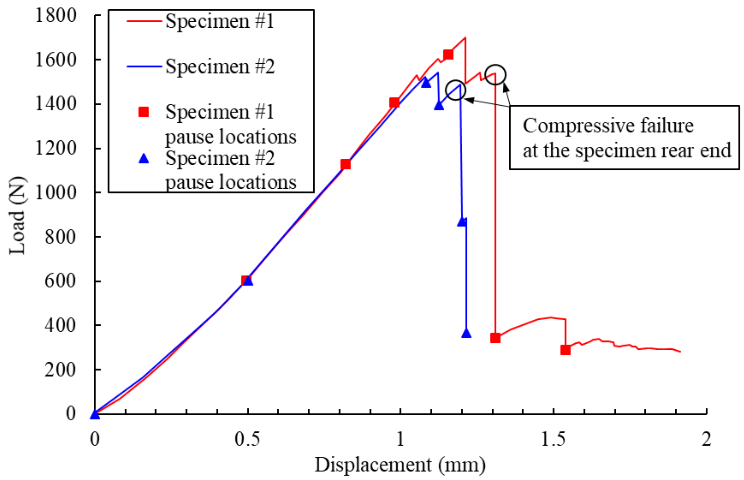

Figure 2. *In situ* CT scan load-displacement curves.

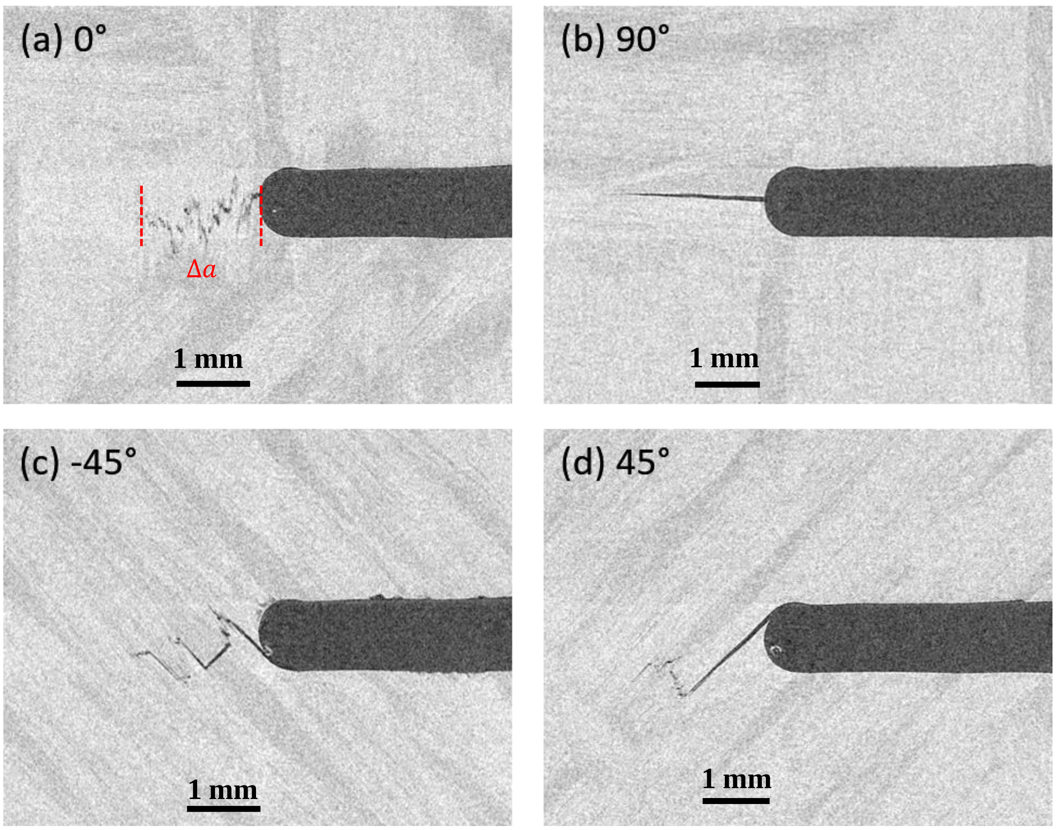

Figure 3. Typical *in situ* CT scan images, taken from Specimen #2 at 1.12 mm displacement (Δa is the effective crack increment).

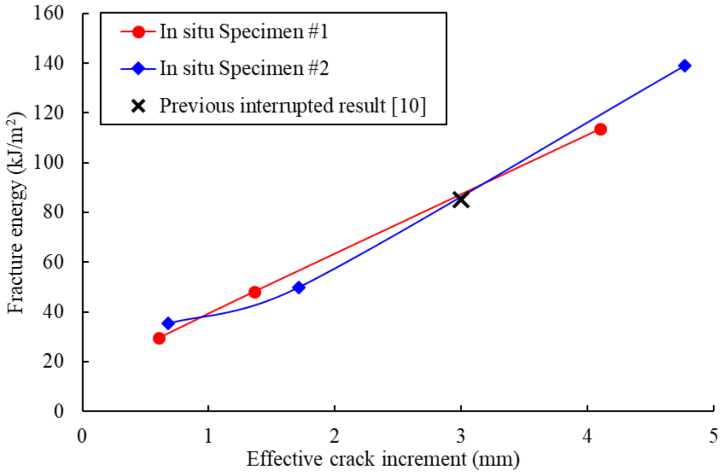

Figure 4. Partial R-curves constructed with *in situ* CT scans.

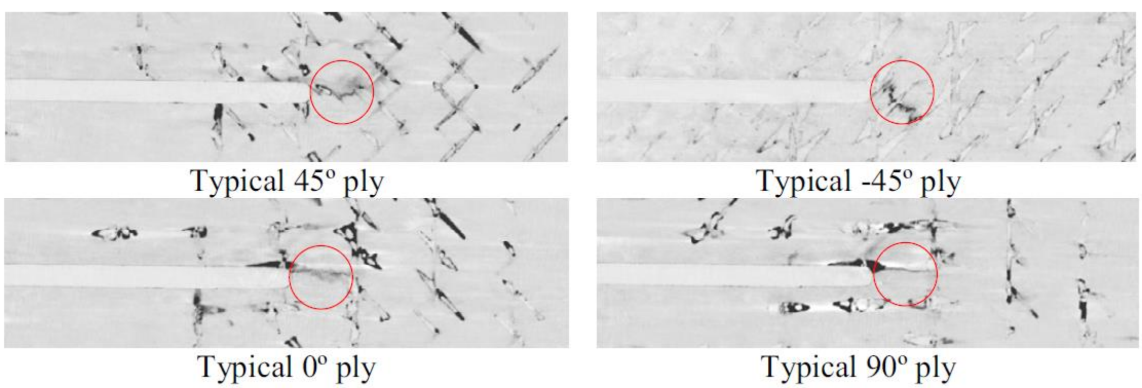

Figure 5. CT scan images with dye penetrant from the previous interrupted OCT test (a red circle indicates a 3 mm diameter) [10].